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A BOLT-ON DEPOSITION SOURCE FOR ULTRA-HIGH-VACUUM GROWTH OF INTERMETALLIC COMPOUND FILMS

by

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An eight-inch ConFlat® flange assembly with both an electron-beam evaporator and a Knudsen cell has been constructed to deposit intermetallic compounds containing transition and group-III metals with specific phase composition. Initial depositions of thin films using this design have shown excellent epitaxy with the desired compound stoichiometry.

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Shop Note: A Bolt-on Deposition Source For Ultra-High Vacuum Growth of Intermetallic Compound Films

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An eight-inch ConFlat® flange assembly with both an electron beam evaporator and a Knudsen cell has been constructed to deposit intermetallic compounds *containing transition* and group III metals with specific phase composition. Initial depositions of thin films using this design have shown excellent epitaxy with the desired compound stoichiometry.

Thin film growth of many intermetallic compounds by molecular beam epitaxy (MBE) techniques is complicated by several factors: the low vapor pressure of transition metals, which requires the use of an electron beam evaporator, the need for precise control of the deposition fluxes to achieve the desired stoichiometry, and the physical constraints of ultra-high vacuum (UHV) chambers. An UHV compatible intermetallic film evaporator (IFE) was designed and fabricated to satisfy these requirements. The compact size of the IFE allows it to be integrated with existing UHV chambers that have an eight-inch ConFlat® flange with line-of-sight to the sample.

The basic design of the IFE employs a modified Thermionics Laboratories Inc. (TLI)[1] water-cooled, rod-fed 3 kW electron beam evaporator and a standard Knudsen cell source, both mounted on a single eight-inch UHV flange. The electron beam evaporator is used for vaporizing species with very high melting points, such as Co, and the Knudsen cell is for lower melting point species, such as Ga or In. The schematic of the IFE is shown in Figure 1. The evaporator utilizes high-purity metal rods of 3/16" diameter, which are available from several commercial suppliers, for evaporation stock, and can be used for several depositions before requiring a chamber vent. The evaporator was modified by replacing the flat boss used for the double sided evaporator seal with a fine-threaded

nut that improves sealing reliability and prevents twisting of the electrical feedthroughs on the evaporator head.

The port accommodating the Knudsen cell assembly is 1.6 inches from the centerline of the 8-inch flange and is set at 11° with respect to a perpendicular to the IFE flange face. The Knudsen cell is mounted on a custom designed 2.75-inch ConFlat® flange that has two mini-nipples; one for a rotary motion drive to operate a shutter and one for a four-pin electrical feedthrough for heater power and a thermocouple. The Knudsen cell assembly was constructed from two pressed BN crucibles.[2] The heat source is constructed of approximately 1 m of 0.25 mm Ta wire wrapped around a threaded 3 cc source crucible, which is snugly inserted into a larger crucible for thermal insulation. The resulting assembly is then heat shielded with Ta foil, mounted on two rods that are tapped into the flange face, and thoroughly degassed under vacuum before use with the IFE apparatus. Typical operating conditions to obtain a crucible temperature of 1000°C are 20 dc volts and 2 amps, with source flux stabilization within 20 minutes.

The integral liquid nitrogen cryoshroud fully encloses both evaporation sources and has a beam containment shield that prevents cross contamination of source materials. The shield also limits the forward and peripheral fields of view of the line-of-sight evaporators. This allows the IFE to operate in chambers that contain sensitive analytical optics. The two source shutters make

determination of both flux rates simple, since they isolate each source from a quartz crystal monitor that can be moved into the actual growth position. Alignment of the sources is straightforward since they share the same vertical focal plane and focal point (in our case the distance from the IFE flange face to the sample is 9.65 inches).

Operation of the IFE in a chamber designed for ultraviolet photoelectron spectroscopy (UPS) with a base pressure of 4 x 10^{-9} Torr yields typical deposition pressures of 1 x 10^{-7} Torr. Rates of material evaporation from the electron beam evaporator are variable and depend on material and applied power[1]. Initial characterization of CoGa intermetallic films grown on GaAs(001) substrates with the IFE show excellent stoichiometry control and single crystal epitaxy[3].

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REFERENCES

1. Thermionics Laboratories Inc., P.O. Box 3711, Hayward, CA. 94540.
2. The R. D. Mathis Co., P.O. Box 6187, Long Beach,CA 90806.
3. Young K. Kim and R. Stanley Williams, to be published.

Figure 1. Schematic of the IFE apparatus and flange layout.

A) Custom 2.75-inch flange for Knudsen cell

B) Standard eight-inch ConFlat® flange

C) Knudsen cell source shutter

D) Beam containment shield

E) Electron beam evaporator shutter

F) Electron beam evaporator head assembly

G) Cryoshroud

H) Rotary feedthrough

I) Double sealing flange assembly

J) Water cooling lines for electron beam heater

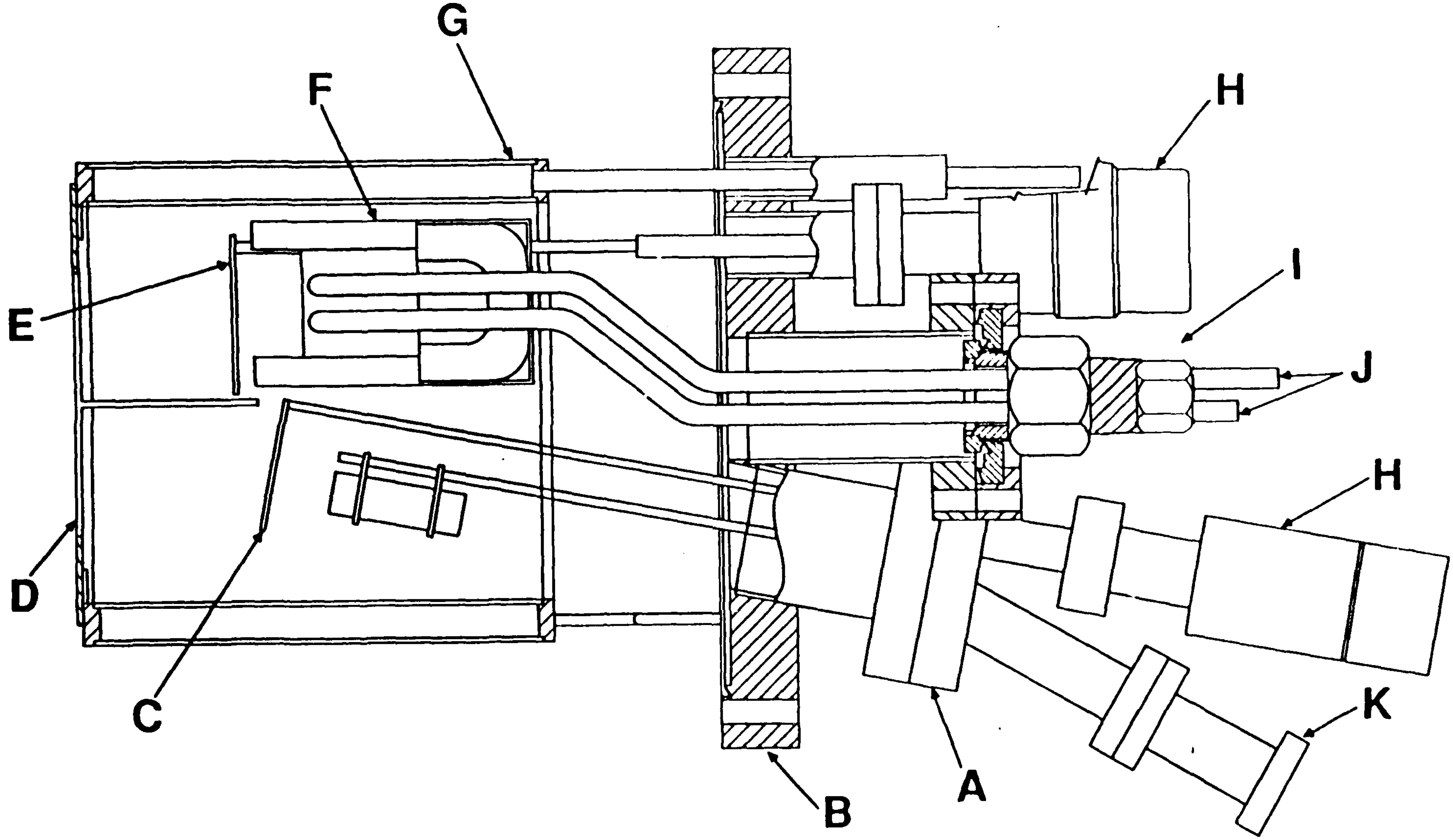
K) Knudsen cell electrical feedthroughs

L) Cryoshroud coolant feedthroughs

M) Electron beam evaporator electrical feedthrough

N) Knudsen cell 2.75-inch port

O) Electron beam evaporator assembly mounted on a 2.75-inch port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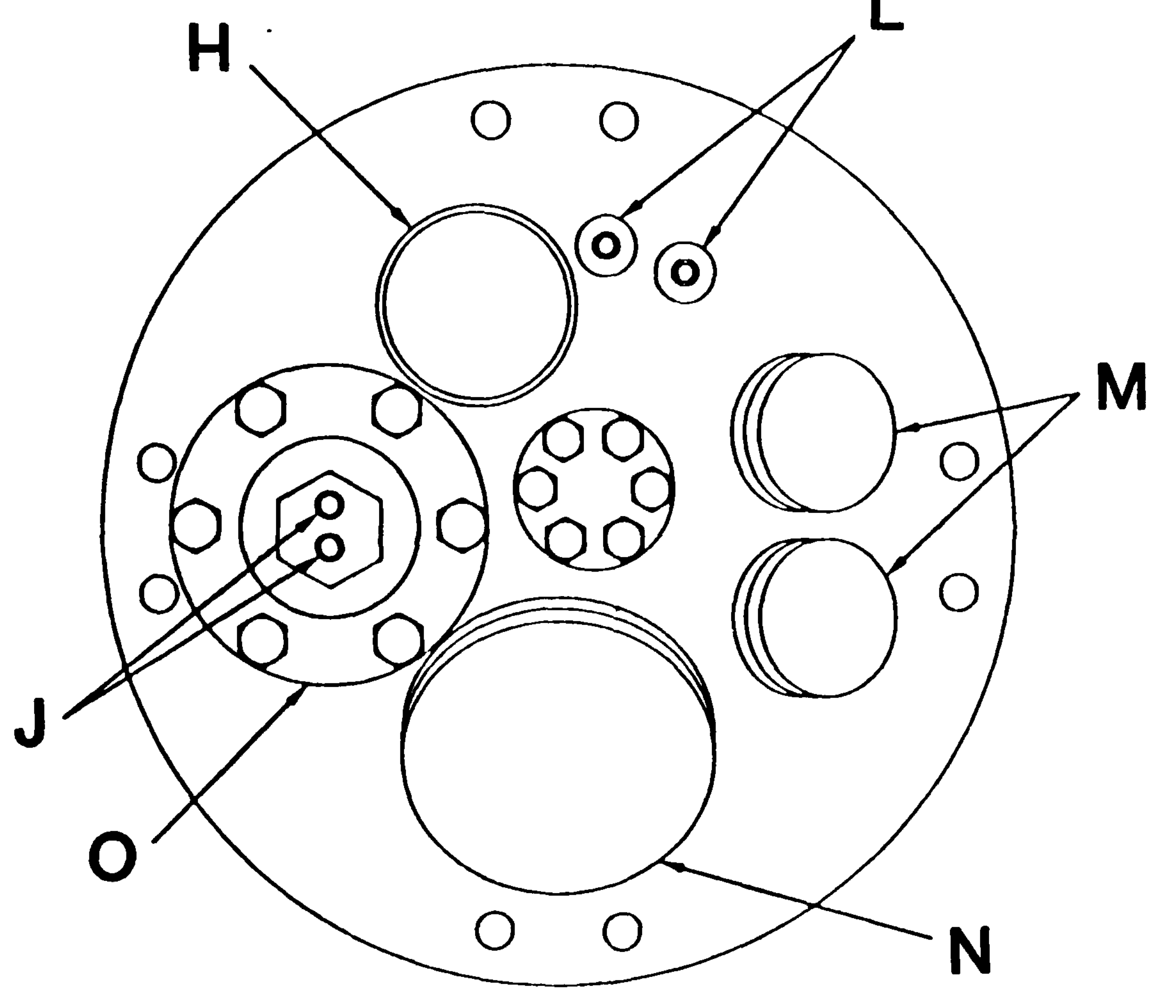
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